TENT COOPERATION TRE. Y

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

To:

Assistant Commissioner for Patents
United States Patent and Trademark Office
Box PCT
Washington, D.C.20231
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 23 June 2000 (23.06.00)	
International application No. PCT/DK99/00559	**Applicant's or agent's file reference** 16544 B PCT
International filing date (day/month/year) 15 October 1999 (15.10.99)	**Priority date** (day/month/year) 04 November 1998 (04.11.98)
Applicant DUER, Victor	

1. The designated Office is hereby notified of its election made:

[X] in the demand filed with the International Preliminary Examining Authority on:

25 May 2000 (25.05.00)

[ ] in a notice effecting later election filed with the International Bureau on:

2. The election [X] was

[ ] was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland

Facsimile No.: (41-22) 740.14.35

Authorized officer

F. Baechler

Telephone No.: (41-22) 338.83.38

1881

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
16544 B PCT		
International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/DK99/00559	15.10.1999	04.11.1998
International Patent Classification (IPC) or national classification and IPC7		
A 23 K 1/00, A 23 K 1/16		
Applicant		
Leo Pharmaceutical Products LTD A/S		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 5 sheets, including this cover sheet.

☒ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 2 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
25.05.2000	05.03.2001
Name and mailing address of the IPEA/SE Patent- och registreringsverket Box 5055 S-102 42 STOCKHOLM Telex 17978 PATOREG-S Facsimile No. 08-667 72 88	Authorized officer Eva Johansson/EÖ Telephone No. 08-782 25 00

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/DK99/00559

I. Basis of the report

1. With regard to the **elements** of the international application:*

☐ the international application as originally filed

☒ the description:
pages 1-17 , as originally filed
pages _____ , filed with the demand
pages _____ , filed with the letter of _____

☒ the claims:
pages _____ , as originally filed
pages _____ , as amended (together with any statement) under article 19
pages _____ , filed with the demand
pages 18-19 , filed with the letter of 05.01.2001

☐ the drawings:
pages _____ , as originally filed
pages _____ , filed with the demand
pages _____ , filed with the letter of _____

☐ the sequence listing part of the description:
pages _____ , as originally filed
pages _____ , filed with the demand
pages _____ , filed with the letter of _____

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.
These elements were available or furnished to this Authority in the following language english which is:

☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).

☒ the language of publication of the international application (under Rule 48.3(b)).

☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

☐ contained in the international application in written form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. ☐ The amendments have resulted in the cancellation of:

☐ the description, pages _____

☐ the claims, Nos. _____

☐ the drawings, sheet/fig _____

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2 (c)).**

* *Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).*

** *Any replacement sheet containing such amendments must be referred to under item I and annexed to this report.*

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/DK99/00559

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims	1-12	YES
	Claims		NO
Inventive step (IS)	Claims		YES
	Claims	1-12	NO
Industrial applicability (IA)	Claims	1-12	YES
	Claims		NO

2. Citations and explanations (Rule 70.7)

The claimed invention relates to a method for the production of animal feed pellets with the addition of a premix, which is a vitamin premix comprising fat/oil and water-soluble vitamins. The surface of the pellets is sprayed with the vitamin premix and the feed pellets are cooled before being sprayed.

New amended claims 1-12 have been filed on 5 January 2001. The auxiliary request claims have been examined.
Claim 1 has been supplemented with the specification of the cooling temperature to be equal or less than 50^0 C, that the vitamin premix also comprises a phytase enzyme and that the pellets are collected in a container.

The technical features "phytase enzyme" and "collected in a container" do not solve the problem of the invention, which is a method for the production of a feed pellets **sprayed** with a vitamin premix and **cooled before spraying.**
It is obvious to a person skilled in the art to chose those products e.g. oil/fat or water soluble vitamins, minerals, amino acids and enzymes that are suitable for each opportunity and mix the products to a liquid premix.
It is also obvious to a person skilled in the art to collect the finished pellets in a container.

.../....

Supplemental Box
(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: V

The following documents are cited in the search report:
D1) EP 689834
D2) EP 231817
D3) EP 682874
D4) ES 2033571
D5) WO 9847389
D6) GB 1457643
D7) RU 2075946 abstract

D2) relates to a method of pelletising animal or human foods with vitamin(s) and similar substances preferably sprinkled onto finished cooled pellets. The pellets are made of foodstuffs or animal feed, mixed and heated in a pelletising process. The pellets are cooled (see col. 7 line 3-15) before the health-promoting additives such as vitamins (see example 3 line 43 and 51-56) are sprayed or rolled on the surface of the pellets (see fig 1, and col. 8).

The claimed invention only differs from the known in the definition of the temperature.
It is obvious to a person skilled in the art from reading the cited document to choose suitable vitamins and other health-promoting additives and to mix these components to a premix and spray the mixture on the pellets at an appropriate temperature. Thus, the claimed invention lacks inventive step.

D4) relates to an animal feed supplement production. Granules are made in a spray tower and then cooled to 35^0 C. The cooled granules are sprayed with a vitamin complex (consisting of different vitamins and other health-promoting additives (see col. 4) which remains fixed to the surface of the granules.

From this document is it known to cool the granules to a temperature of 35^0 C before spraying the mixture on the granules. Thus, it is obvious to a person from reading the cited documents to cool granules or pellets before spraying with a mixture.

D1) relates to a food and feed supplement comprising vitamins. A core mixture of vitamins is prepared. A basic mixture is prepared and agglomerated. After drying granules are made and sprayed with the core mixture. The core mixture contains vitamins and other health-promoting additives (see example 5 page 11-12).

..../...

Supplemental Box
(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: V

D3) relates to bioactive feed pellets comprising besides commonly used nutritionally valuable components, a bioactive ingredient such as therapeutically or prophylactically active compound, a vaccine, a pigment, a vitamin, and/or an enzyme. The bioactive ingredient is applied to the pellets in the form of a primary coated dispersion.

D5), D6) and D7) relate to among other things a vitamin premix comprising commonly used vitamins and other health-promoting additives, see particular page 7 line 15 to page 8 line 14 in D5) and example 1-5 in D6).

From the last cited documents it is known to prepare mixtures containing e.g. oil/fat and water- soluble vitamins, minerals, amino acids and enzymes.
According to what is known from the cited documents is it obvious to a person skilled in the art to mix suitable vitamins with an enzyme.
There is no information in the application that an addition of a phytase enzyme in the vitamin premix would have any special effect on the spraying process.

The claimed invention is considered to be novel and has industrial applicability but is considered to lack inventive step.

CLAIMS

1. Method for the production of animal feed pellets with the addition of a premix, characterized in that the premix is a vitamin premix comprising fat/oil- and water-soluble vitamins, that the surface of the feed pellets is sprayed with the vitamin premix, and in that the feed pellets are subjected to cooling before being sprayed.

2. Method according to claim 1, characterized in that after being sprayed the feed pellets are collected in a container.

3. Method according to claim 1 and 2, characterized in that the vitamin premix is formulated as a function of animal species.

4. Method according to any of the foregoing claims, characterized in that the feed pellets pass a rotor-spray/rotor nozzle when being sprayed.

5. Method according to any of the foregoing claims, characterized in that the feed pellets are also sprayed with a solution comprising minerals.

6. Method according to any of the foregoing claims, characterized in that the vitamin premix also comprises amino acids dissolved in said vitamin premix.

7. Method according to any of the foregoing claims, characterized in that the vitamin premix also comprises digestibility-promoting enzymes dissolved in said vitamin premix.

8. Method according to any of the foregoing claims, characterized in that the vitamin premix also comprises a phytase enzyme dissolved in said vitamin premix.

9. Method for the mixing a vitamin premix comprising fat/oil-soluble vitamins, characterized in that the premix also comprises water-soluble vitamins, that the water phase comprises propylene glycol and EDTA and nicotinamide, after which a B_2 vitamin is subsequently added such as riboflavin and thereafter sodium hydrochloride (NaOH).

10. Method according to claim 9, characterized in that carbamide/urea is added before the addition of the B_2 vitamin.

11. Method according to claim 8, 9 and 10, characterized in that hydrochloric acid (HCl) is also added, and that further B vitamins are subsequently added, mainly biotin and pyridoxine hydrochloride.

12. Method according to claim 9, 10 and 11, characterized in that the oil phase comprises A, D and E vitamins, a solubilisator and also antioxidants, the mixing of which is carried out at a temperature interval of around 50-70°, preferably at around 60°.

13. Method according to claims 9-12, characterized in that the oil phase and the water phase are mixed together while being stirred, and that the temperature of the water phase is 35-45°C.

14. Method according to claims 9-13, characterized in that a phytase enzyme is added to the vitamin premix, said premix preferably having a temperature of 20-30°C.

15. Vitamin premix comprising soluble vitamins, characterized in that the oil-soluble vitamins comprise A, D and E vitamins, and that the premix also comprises water-soluble vitamins such as K vitamin, C vitamin and various B vitamins.

PCT

REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No.

International Filing Date

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference *(if desired) (12 characters maximum)* 16544 B PCT

Box No. I TITLE OF INVENTION METHOD FOR THE PRODUCTION OF FEED PELLETS, MIX OF A PREMIX AND VITAMIN PREMIX.

Box No. II APPLICANT

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

Løvens Kemiske Fabrik
Leo Pharmaceutical Products
Industriparken 55
DK-2750 Ballerup
Denmark

☐ This person is also inventor.

Telephone No.

Facsimile No.

Teleprinter No.

State *(that is, country)* of nationality: DK

State *(that is, country)* of residence: DK

This person is applicant for the purposes of: ☐ all designated States ☒ all designated States except the United States of America ☐ the United States of America only ☐ the States indicated in the Supplemental Box

Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

DUER, Victor
Højbovænge 61
DK-3500 Værløse
Denmark

This person is:

☐ applicant only

☒ applicant and inventor

☐ inventor only *(If this check-box is marked, do not fill in below.)*

State *(that is, country)* of nationality: DK

State *(that is, country)* of residence: DK

This person is applicant for the purposes of: ☐ all designated States ☐ all designated States except the United States of America ☒ the United States of America only ☐ the States indicated in the Supplemental Box

☐ Further applicants and/or (further) inventors are indicated on a continuation sheet.

Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: ☒ agent ☐ common representative

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

LARSEN & BIRKEHOLM A/S
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Denmark

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Teleprinter No.

☐ Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

See Notes to the request form

EL243104167US

Box No.V DESIGNATION OF STATES

The following designations are hereby made under Rule 4.9(a) *(mark the applicable check-boxes; at least one must be marked)*:

Regional Patent

- [X] AP ARIPO Patent: GH Ghana, GM Gambia, KE Kenya, LS Lesotho, MW Malawi, SD Sudan, SL Sierra Leone, SZ Swaziland, UG Uganda, ZW Zimbabwe, and any other State which is a Contracting State of the Harare Protocol and of the PCT
- [X] EA Eurasian Patent: AM Armenia, AZ Azerbaijan, BY Belarus, KG Kyrgyzstan, KZ Kazakhstan, MD Republic of Moldova, RU Russian Federation, TJ Tajikistan, TM Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT
- [X] EP European Patent: AT Austria, BE Belgium, CH and LI Switzerland and Liechtenstein, CY Cyprus, DE Germany, DK Denmark, ES Spain, FI Finland, FR France, GB United Kingdom, GR Greece, IE Ireland, IT Italy, LU Luxembourg, MC Monaco, NL Netherlands, PT Portugal, SE Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT
- [ ] OA OAPI Patent: BF Burkina Faso, BJ Benin, CF Central African Republic, CG Congo, CI Côte d'Ivoire, CM Cameroon, GA Gabon, GN Guinea, GW Guinea-Bissau, ML Mali, MR Mauritania, NE Niger, SN Senegal, TD Chad, TG Togo, and any other State which is a member State of OAPI and a Contracting State of the PCT *(if other kind of protection or treatment desired, specify on dotted line)* .....

National Patent *(if other kind of protection or treatment desired, specify on dotted line)*:

- [ ] AE United Arab Emirates
- [ ] AL Albania .....
- [ ] AM Armenia .....
- [X] AT Austria and Utility Model ....
- [X] AU Australia .....
- [ ] AZ Azerbaijan
- [ ] BA Bosnia and Herzegovina .....
- [ ] BB Barbados
- [X] BG Bulgaria .....
- [X] BR Brazil .....
- [X] BY Belarus .....
- [X] CA Canada
- [X] CH and LI Switzerland and Liechtenstein
- [X] CN China .....
- [ ] CU Cuba .....
- [X] CZ Czech Republic and Utility Model
- [X] DE Germany and Utility Model
- [X] DK Denmark and Utility Model
- [X] EE Estonia and Utility Model
- [ ] ES Spain .....
- [X] FI Finland and Utility Model
- [X] GB United Kingdom
- [X] GD Grenada
- [X] GE Georgia .....
- [X] GH Ghana .....
- [X] GM Gambia
- [X] HR Croatia .....
- [X] HU Hungary .....
- [X] ID Indonesia
- [X] IL Israel .....
- [X] IN India .....
- [ ] IS Iceland
- [ ] JP Japan .....
- [ ] KE Kenya .....
- [ ] KG Kyrgyzstan .....
- [X] KP Democratic People's Republic of Korea ....
  .....
- [X] KR Republic of Korea .....
- [X] KZ Kazakhstan .....
- [X] LC Saint Lucia
- [X] LK Sri Lanka
- [X] LR Liberia
- [X] LS Lesotho .....
- [X] LT Lithuania
- [X] LU Luxembourg
- [X] LV Latvia
- [X] MD Republic of Moldova .....
- [ ] MG Madagascar .....
- [ ] MK The former Yugoslav Republic of Macedonia ...
- [x] MA Morroco .....
- [X] MN Mongolia
- [X] MW Malawi .....
- [X] MX Mexico .....
- [X] NO Norway
- [X] NZ New Zealand .....
- [X] PL Poland .....
- [X] PT Portugal .....
- [X] RO Romania
- [X] RU Russian Federation .....
- [X] SD Sudan
- [X] SE Sweden
- [X] SG Singapore
- [X] SI Slovenia .....
- [X] SK Slovakia and Utility Model
- [X] SL Sierra Leone .....
- [X] TJ Tajikistan .....
- [X] TM Turkmenistan .....
- [X] TR Turkey .....
- [X] TT Trinidad and Tobago .....
- [X] UA Ukraine .....
- [X] UG Uganda .....
- [ ] US United States of America .....
- [x] TZ United Rep. of Tanzania .....
- [ ] UZ Uzbekistan .....
- [X] VN Viet Nam .....
- [X] YU Yugoslavia .....
- [X] ZA South Africa .....
- [X] ZW Zimbabwe .....

Check-boxes reserved for designating States which have become party to the PCT after issuance of this sheet:

- [ ] CR Costa Rica .....
- [ ] DM Dominica .....

Precautionary Designation Statement: In addition to the designations made above, the applicant also makes under Rule 4.9(b) all other designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. *(Confirmation of a designation consists of the filing of a notice specifying that designation and the payment of the designation and confirmation fees. Confirmation must reach the receiving Office within the 15-month time limit.)*

Box No. VI PRIORITY CLAIM ☐ Further priority claims are indicated in the Supplemental Box.

Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is: national application: country	regional application: regional Office	international application: receiving Office
item (1) 4 November 1998 (04.11.98)	PA 1998 01422	DK		
item (2) 18 January 1999 (18.01.99)	PA 1999 00055	DK		
item (3)				

☒ The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) *(only if the earlier application was filed with the Office which for the purposes of the present international application is the receiving Office)* identified above as item(s): 1) + 2)

* *Where the earlier application is an ARIPO application, it is mandatory to indicate in the Supplemental Box at least one country party to the Paris Convention for the Protection of Industrial Property for which that earlier application was filed (Rule 4.10(b)(ii)). See Supplemental Box.*

Box No. VII INTERNATIONAL SEARCHING AUTHORITY

Choice of International Searching Authority (ISA) *(if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used)*: ISA / SE

Request to use results of earlier search; reference to that search *(if an earlier search has been carried out by or requested from the International Searching Authority)*: Date *(day/month/year)* | Number | Country *(or regional Office)*

Box No. VIII CHECK LIST; LANGUAGE OF FILING

This international application contains the following number of sheets:

request	:	3
description (excluding sequence listing part)	:	17
claims	:	3
abstract	:	1
drawings	:	
sequence listing part of description	:	
Total number of sheets	:	24

This international application is accompanied by the item(s) marked below:

1. ☒ fee calculation sheet
2. ☐ separate signed power of attorney
3. ☐ copy of general power of attorney; reference number, if any:
4. ☐ statement explaining lack of signature
5. ☐ priority document(s) identified in Box No. VI as item(s):
6. ☐ translation of international application into (language):
7. ☐ separate indications concerning deposited microorganism or other biological material
8. ☐ nucleotide and/or amino acid sequence listing in computer readable form
9. ☐ other *(specify)*:

Figure of the drawings which should accompany the abstract:

Language of filing of the international application: Danish

Box No. IX SIGNATURE OF APPLICANT OR AGENT

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).

Copenhagen, 15 October 1999
LARSEN & BIRKEHOLM A/S
Skandinavisk Patentbureau

Tenna M. Pedersen

For receiving Office use only

1. Date of actual receipt of the purported international application:
2. Drawings: ☐ received: ☐ not received:
3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:
4. Date of timely receipt of the required corrections under PCT Article 11(2):
5. International Searching Authority (if two or more are competent): ISA /
6. ☐ Transmittal of search copy delayed until search fee is paid.

For International Bureau use only

Date of receipt of the record copy by the International Bureau:

RECORD COPY PCT

REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No. PCT/DK 99/00559

International Filing Date RO/DK 15 OCTOBER 1999

Patentdirektoratet
Danish Patent Office
PCT International Application

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference *(if desired) (12 characters maximum)* 16544 B PCT

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Løvens Kemiske Fabrik
Leo Pharmaceutical Products Ltd. A/S
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Denmark

☐ This person is also inventor.

Telephone No.

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State *(that is, country)* of residence: DK

This person is applicant for the purposes of: ☐ all designated States ☒ all designated States except the United States of America ☐ the United States of America only ☐ the States indicated in the Supplemental Box

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DUER, Victor
Højbovænge 61
DK-3500 Værløse
Denmark

This person is:

☐ applicant only

☒ applicant and inventor

☐ inventor only *(If this check-box is marked, do not fill in below.)*

State *(that is, country)* of nationality: DK

State *(that is, country)* of residence: DK

This person is applicant for the purposes of: ☐ all designated States ☐ all designated States except the United States of America ☒ the United States of America only ☐ the States indicated in the Supplemental Box

☐ Further applicants and/or (further) inventors are indicated on a continuation sheet.

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Teleprinter No.

☐ **Address for correspondence:** Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

Box No.V DESIGNATION OF STATES

The following designations are hereby made under Rule 4.9(a) *(mark the applicable check-boxes; at least one must be marked)*:

Regional Patent

- [X] **AP** ARIPO Patent: GH Ghana, GM Gambia, KE Kenya, LS Lesotho, MW Malawi, SD Sudan, SL Sierra Leone, SZ Swaziland, UG Uganda, ZW Zimbabwe, and any other State which is a Contracting State of the Harare Protocol and of the PCT
- [X] **EA** Eurasian Patent: AM Armenia, AZ Azerbaijan, BY Belarus, KG Kyrgyzstan, KZ Kazakhstan, MD Republic of Moldova, RU Russian Federation, TJ Tajikistan, TM Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT
- [X] **EP** European Patent: AT Austria, BE Belgium, CH and LI Switzerland and Liechtenstein, CY Cyprus, DE Germany, DK Denmark, ES Spain, FI Finland, FR France, GB United Kingdom, GR Greece, IE Ireland, IT Italy, LU Luxembourg, MC Monaco, NL Netherlands, PT Portugal, SE Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT
- [X] **OA** OAPI Patent: BF Burkina Faso, BJ Benin, CF Central African Republic, CG Congo, CI Côte d'Ivoire, CM Cameroon, GA Gabon, GN Guinea, GW Guinea-Bissau, ML Mali, MR Mauritania, NE Niger, SN Senegal, TD Chad, TG Togo, and any other State which is a member State of OAPI and a Contracting State of the PCT *(if other kind of protection or treatment desired, specify on dotted line)* .....

National Patent *(if other kind of protection or treatment desired, specify on dotted line)*:

- [X] AE United Arab Emirates
- [X] AL Albania .....
- [X] AM Armenia .....
- [X] AT Austria and Utility Model .....
- [X] AU Australia .....
- [X] AZ Azerbaijan
- [X] BA Bosnia and Herzegovina .....
- [X] BB Barbados
- [X] BG Bulgaria .....
- [X] BR Brazil .....
- [X] BY Belarus .....
- [X] CA Canada
- [X] CH and LI Switzerland and Liechtenstein
- [X] CN China .....
- [X] CU Cuba .....
- [X] CZ Czech Republic and Utility Model
- [X] DE Germany and Utility Model
- [X] DK Denmark and Utility Model
- [X] EE Estonia and Utility Model
- [X] ES Spain .....
- [X] FI Finland and Utility Model
- [X] GB United Kingdom
- [X] GD Grenada
- [X] GE Georgia .....
- [X] GH Ghana .....
- [X] GM Gambia
- [X] HR Croatia .....
- [X] HU Hungary .....
- [X] ID Indonesia
- [X] IL Israel .....
- [X] IN India .....
- [X] IS Iceland
- [X] JP Japan .....
- [X] KE Kenya .....
- [X] KG Kyrgyzstan .....
- [X] KP Democratic People's Republic of Korea .....
- [X] KR Republic of Korea .....
- [X] KZ Kazakhstan .....
- [X] LC Saint Lucia
- [X] LK Sri Lanka
- [X] LR Liberia
- [X] LS Lesotho .....
- [X] LT Lithuania
- [X] LU Luxembourg
- [X] LV Latvia
- [X] MD Republic of Moldova .....
- [X] MG Madagascar .....
- [X] MK The former Yugoslav Republic of Macedonia ...
- [X] MA Morroco .....
- [X] MN Mongolia
- [X] MW Malawi .....
- [X] MX Mexico .....
- [X] NO Norway
- [X] NZ New Zealand .....
- [X] PL Poland .....
- [X] PT Portugal .....
- [X] RO Romania
- [X] RU Russian Federation .....
- [X] SD Sudan
- [X] SE Sweden
- [X] SG Singapore
- [X] SI Slovenia .....
- [X] SK Slovakia and Utility Model
- [X] SL Sierra Leone .....
- [X] TJ Tajikistan .....
- [X] TM Turkmenistan .....
- [X] TR Turkey .....
- [X] TT Trinidad and Tobago .....
- [X] UA Ukraine .....
- [X] UG Uganda .....
- [X] US United States of America .....
- [X] TZ United Rep. of Tanzania
- [X] UZ Uzbekistan .....
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Precautionary Designation Statement: In addition to the designations made above, the applicant also makes under Rule 4.9(b) all other designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. *(Confirmation of a designation consists of the filing of a notice specifying that designation and the payment of the designation and confirmation fees. Confirmation must reach the receiving Office within the 15-month time limit.)*

Box No. VI PRIORITY CLAIM ☐ Further priority claims are indicated in the Supplemental Box.

Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is: national application: country	regional application:* regional Office	international application: receiving Office
item (1) 4 November 1998 (04.11.98)	PA 1998 01422	DK		
item (2) 18 January 1999 (18.01.99)	PA 1999 00055	DK		
item (3)				

☒ The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) *(only if the earlier application was filed with the Office which for the purposes of the present international application is the receiving Office)* identified above as item(s): 1) + 2)

* *Where the earlier application is an ARIPO application, it is mandatory to indicate in the Supplemental Box at least one country party to the Paris Convention for the Protection of Industrial Property for which that earlier application was filed (Rule 4.10(b)(ii)). See Supplemental Box.*

Box No. VII INTERNATIONAL SEARCHING AUTHORITY

Choice of International Searching Authority (ISA) *(if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used)* : ISA / SE

Request to use results of earlier search; reference to that search *(if an earlier search has been carried out by or requested from the International Searching Authority)*: Date *(day/month/year)* Number Country *(or regional Office)*

Box No. VIII CHECK LIST; LANGUAGE OF FILING

This international application contains the following **number of sheets**:

request	:	3
description (excluding sequence listing part)	:	17
claims	:	3
abstract	:	1
drawings	:	
sequence listing part of description	:	
Total number of sheets	:	24

This international application is **accompanied by** the item(s) marked below:

1. ☒ fee calculation sheet
2. ☐ separate signed power of attorney
3. ☐ copy of general power of attorney; reference number, if any:
4. ☐ statement explaining lack of signature
5. ☐ priority document(s) identified in Box No. VI as item(s):
6. ☐ translation of international application into (language):
7. ☐ separate indications concerning deposited microorganism or other biological material
8. ☐ nucleotide and/or amino acid sequence listing in computer readable form
9. ☐ other *(specify)*:

Figure of the drawings which should accompany the abstract:

Language of filing of the international application: Danish

Box No. IX SIGNATURE OF APPLICANT OR AGENT

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).

Copenhagen, 15 October 1999
LARSEN & BIRKEHOLM A/S
Skandinavisk Patentbureau

Tenna M. Pedersen

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1. Date of actual receipt of the purported international application: RO/DK 15 OCTOBER 1999 (15.10.99)
2. Drawings: ☐ received: ☐ not received:
3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:
4. Date of timely receipt of the required corrections under PCT Article 11(2):
5. International Searching Authority (if two or more are competent): ISA / SE
6. ☐ Transmittal of search copy delayed until search fee is paid.

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Date of receipt of the record copy by the International Bureau: 01 NOVEMBER 1999 (01.11.99)

PCT/DK 99/00559

FREMGANGSMÅDE TIL FREMSTILLING AF FODERPILLER, BLANDING AF EN FORBLANDING SAMT VITAMINFORBLANDING

Opfindelsen angår en fremgangsmåde til fremstilling af foderpiller til dyr tilsat en forblanding.

Opfindelsen angår tillige en fremgangsmåde til blanding af en vitaminforblanding omfattende fedt/olieopløselige vitaminer.

Endelig omfatter opfindelsen en vitaminforblanding omfattende opløselige vitaminer.

Fra EP-A-682874 er det kendt at tilføre dyrefoder vitaminer, hvilke vitaminer befinder sig i en fedt- eller olieopløsning, ved hvilket produkt og ved hvilken metode der skulle kunne opstå en mindre risiko for dekomponering af vitamintilsætningen. Skriftet angiver dog ikke en metode til tilsætning af det totale fysiologiske vitaminbehov, der måtte være i forbindelse med dyrefoder, ligesom skriftet ej heller angiver en metode, hvorved denne tilsætning af vitaminer foregår under forhold, hvor en ødelæggelse og dekomponering af de enkelte vitaminer, det være sig både de fedt- og vandopløselige, minimeres mest muligt.

Det er formålet med nærværende opfindelse at tilvejebringe en fremgangsmåde, hvor ovennævnte problemer imødegås, og hvor genfindingsprocenten for de tilsatte vitaminer er meget høj, hvorfor overdosering undgås, og hvor det er muligt at foretage en påsprøjtning af flydende forblandinger på foderpiller, således at det overflødiggøres at investere i større anlæg til håndtering af forblandinger efter tørtilsætningsmetoden.

Det er ved denne metode således tillige formålet at sikre, at proces- og lagerstabiliteten optimeres, således at der foregår mindst mulig dekomponering af vitaminerne, efter at disse er påsprøjtet det industrielt fremstillede foder.

Dette formål opnås med en fremgangsmåde af den i indledningen angivne art, og hvor tillige forblandingen er en vitaminforblanding omfattende fedt/olie- og vandopløselige vitaminer, hvor foderpillerne på overfladen påsprøjtes med vitaminforblandingen, samt hvor foderpillerne inden påsprøjtningen har undergået en afkøling.

Ved at foretage en påsprøjtning af foderpillerne, når disse er fremstillet, og ved tillige at foretage en passende afkøling til maksimalt 50°C og et optimalt temperaturniveau på 25-35°C, er det muligt at foretage en efterfølgende påsprøjtning på disses overflade af vitaminforblandingen, som omfatter såvel fedt/olie- som vandopløselige vitaminer, hvorved sikres, at foderpillerne får et optimalt indhold af vitaminer til den pågældende dyreart, det være sig grise eller kyllinger m.v. Der opnås ved denne metode en langt højere genfinding af de tilsatte vitaminer end ved tilsvarende dosering via en tørret vitaminforblanding.

Den usædvanligt forbedrede genfindingsprocent for vitaminerne efter påsprøjtning har betydning for dyrenes vitaminforsyning og er således også betydningsfuld i relation til offentlig foderstofkontrol. Det er således væsentligt, at vitamindoseringen ved flydende påsprøjtning kan reduceres uden at reducere dyrenes forsyning af tilsatte vitaminer.

Ved at tilvejebringe en fremgangsmåde ifølge opfindelsen, og som yderligere angivet i krav 2, opnås, at foderpillerne opbevares ved en relativ lav iltkoncentration, hvorved nedbrydningen minimeres og lagerstabiliteten

optimeres. En lagertid på 2-6 uger, hvilket dog er en relativ kort men realistisk lagertid, bevirker således, at vitaminstabiliteten og genfindingen er overraskende stor.

Ved at tilvejebringe en fremgangsmåde ifølge opfindelsen, og som yderligere angivet i krav 3, etableres mulighed for at regulere de færdig-fremstillede foderpiller med den påsprøjtede vitaminforblanding, alt efter hvilket dyr der måtte være tale om. Det vil med andre ord sige, at hvor eksempelvis foderet skal bruges til kyllinger, kan der påsprøjtes vitaminer omfattende C-vitamin.

Ved at tilvejebringe en fremgangsmåde ifølge opfindelsen, og som yderligere angivet i krav 4, fås en god påsprøjtning på de enkelte piller, således at et hvilket som helst udsnit af pillerne vil give anledning til i det væsentlige den samme koncentrationsmængde af den påsprøjtede forblanding.

Ved at tilvejebringe en fremgangsmåde ifølge opfindelsen, og som yderligere angivet i krav 5, opnås, at foderpillerne tillige kommer til at omfatte de mineraler, som måtte være relevante i relation til de enkelte dyr.

Ved at tilvejebringe en fremgangsmåde ifølge opfindelsen, og som yderligere angivet i krav 6, opnås mulighed for at tilsætte yderligere næringsstoffer, såsom lysin, mhehionin, threonin, leucin og isoleucin.

Ved at tilvejebringe en fremgangsmåde ifølge opfindelsen, og som yderligere angivet i krav 7, opnås mulighed for enzymtilsætning, såsom kulhydrat- og proteinspaltede enzymer.

Ved at tilvejebringe en fremgangsmåde ifølge opfindelsen, og som yderligere angivet i krav 8, opnås en besparelse i tilsat fosformængde, og hvil-

ket i øvrigt bevirker, at det valgte B-vitamin overvejende bør være riboflavin og ikke natriumriboflavinfosfat.

Opfindelsen angår også en fremgangsmåde til blanding af selve den vitaminforblanding, der benyttes under påsprøjtning, som angivet i indledningen, og hvor forblandingen tillige omfatter vandopløselige vitaminer, at vandfasen omfatter propylenglycol samt EDTA og nicotinamid, hvorefter der efterfølgende tilsættes et B_2-vitamin som riboflavin og natriumhydroxid (NaOH).

Ved en sådan forblanding omfattende de nævnte komponenter opnås, at det er muligt at opløse riboflavin, hvilket er vigtigt, idet riboflavin dels er prismæssigt fordelagtig dels nødvendig at bruge som B_2-vitamin, hvis der også sidenhen skal tilsættes fytaseenzym.

Ved at tilvejebringe en fremgangsmåde ifølge opfindelsen, og som yderligere angivet i krav 10, opnås, at det er muligt at tilsætte større mængder af B_2-vitamin som riboflavin.

Ved at tilvejebringe en fremgangsmåde ifølge opfindelsen, og som yderligere angivet i krav 11, opnås en sænkning af pH, således at den tilsatte riboflavin eller natriumriboflavinfosfat ikke ødelægges af den opnåede høje pH, som etableres ved tilsætning af natriumhydroxid (NaOH).

Ved at tilvejebringe en fremgangsmåde ifølge opfindelsen, og som yderligere angivet i krav 12, opnås en hensigtsmæssig sammensætning af oliefasen.

Ved at tilvejebringe en fremgangsmåde ifølge opfindelsen, og som yderligere angivet i krav 13, opnås en homogen substans af forblandingen under omrøring.

Ved at tilvejebringe en fremgangsmåde ifølge opfindelsen, og som yderligere angivet i krav 14, opnås, at mængden af udskilt fosfat fra dyrene reduceres. Desuden er riboflavinen sikret opløselighed grundet ureas tilstedeværelse.

Temperaturen på 20-30° i forblandingen kan opnås ved tilblanding af koldt ledningsvand forud for tilsætning af fytasen.

Opfindelsen angår endvidere en vitaminforblanding, således som denne ses angivet i krav 15.

Opfindelsen vil blive forklaret nærmere under henvisning til nedenstående eksempel.

FLYDENDE VITAMINER:

Kode:	**Råvare:**	**Vægt i gram:**	
01-A	Vitamin-A-acetat 2,5 mio IU/g	0,930	Oliefase
02-A	Vitamin-D_3-olie 4,0 mio IU/g	0,060	-
03-A	Vitamin-E-olie 97%	39,950	-
04-A	Ethoxyquin	2,000	-
05-A	Bredol 694	50,000	-
01-B	Cholinklorid 75%-opløsning*	0	Vandfase I
02-B	Vitamin-B_{12} 2%-opløsning	0,610	-

K de:	Råvar :	Vægt i gram	
03-B	Panthenol	5,100	-
04-B	Biotin 2% til flydende vitaminer	1,040	-
05-B	Pyridoxinhydroklorid	1,240	-
06-B	Natriumriboflavinfosfat	0,590	-
07-B	Natriumhydroxid-opløsning 34° Bé	2,600	-
08-B	Riboflavin 97%	0,590	-
09-B	Saltsyre 10%	2,800	
09-B	Folinsyre 100%*	0	-
10-B	Urea 46 (Carbamid)	50,000	-
11-B	Nicotinamid (=niacinamid)	9,050	-
12-B	Kaliumsorbat (konserveringsmiddel)	2,000	-
13-B	Tetracemindinatrium (komplexbinder)	2,000	-
14-B	Propylenglycol (opløseligheds-fremmende samt konserverings-middel)	100,000	-
15-B	Ledningsvand	666,550	-
01-C	Fytase-opløsning 5000 FTU/g	60,000	Vandfase II
02-C	Thiaminhydroklorid	0,990	-
03-C	Menadionnatriumbisulfit 100%	1,900	-
	I alt, gram:	1000,000	

* er relevante ved fjerkræ.

Før basen 07 tilsættes, er pH ca. 6, hvorefter basetilsætningen foranlediger en pH-stigning til ca. 10-11, hvilket er nødvendigt for, at riboflavinen (08)

kan opløses. Efterfølgende tilsættes komponent 09 for at foretage en sænkning af pH til omkring 8, idet stoffet 08 ødelægges ved en for høj pH. Ved at foretage en opblanding som beskrevet opnås, at alle vitaminer er til stede i de ønskede koncentrationer, og uden at der foregår en destruktion af disse.

Generelt vil carbamid vægtprocentintervallet af den totale mængde ligge i intervallet 2-10%, fortrinsvis omkring 5%.

Hvis natriumriboflavinfosfat udelades, vil den nødvendige mængde carbamid i eksemplet stige til 50-60 gram for at sikre riboflavinens opløselighed.

Selve opløsningen bestående af såvel vand- som olieopløselige vitaminer påsprøjtes efterfølgende foderpiller til dyr. Denne påsprøjtning kan foregå, før pillerne opbevares i siloer, eller påsprøjtning kan foregå, når pillerne læsses på en transportcontainer. Begge løsninger muliggør en differentiering mellem de forskellige dyr, f.eks. grise kontra kyllinger, samt differentiering ifølge kundeønsker.

Det har overraskende vist sig, at når der foretages denne påsprøjtning på vitaminerne, foregår der ikke nogen destruktion eller udfældning af vitaminerne, hvorfor pillerne er at betragte som særdeles stabile. Årsagen til, at der ikke foregår nogen destruktion, kan eventuelt være, at pillerne opbevares i siloer og containere, hvor der er en lav iltspænding.

Fremstillingsprincip

Det endelige præparat består af en oliefase omfattende de såkaldt fedtopløselige vitaminer opblandet i en afpasset mængde af en egnet solubilisator; her anvendt Bredol 694. Denne blanding, forud opvarmet til 55-65°C,

hældes/pumpes i afpasset strøm ned i vandfasen indeholdende de vandopløselige komponenter og med fastlagt temperatur på 40-45°C. Fordelingen af oliefasen med efterfølgende opløsning i vandfasen (- den egentlige solubiliseringsproces) skal foregå under konstant omrøring og i et passende roligt tempo.

Blandeprocedure

Oliefasen (kode: A). Hjælpestoffet 05 tilsættes enkeltvist og i anførte rækkefølge, vitaminerne 01, 02 og 03 samt antioxidanten 04. Komponenterne er forvarmet for at opnå en egnet konsistens ved håndteringen. Der blandes til ensartethed. Blandingen vil herefter fortsat være homogen.

Vandfasen I (kode: B). Afmålt mængde ledningsvand 15 (forvarmet til ca. 45°C) tilblandes 14. Heri opløses i nævnte rækkefølge 13, 12, 11 og 10. Derefter tilblandes 08 (- uopløselig i vand på dette procestrin). Når stoffet fremstår ensartet fordelt i vandfasen, tilsættes basen 07. Efter endt opløsning af 08 tilsættes syren 09 og derefter enkeltvis og i nævnte rækkefølge 06, 05, 04, 03 samt 02. Produktet fremstår som en let opaliserende væske.

Solubiliseringstrin (A ⇒ B): Oliefasen A pumpes nu i en tynd og uafbrudt stråle ned i vandfase I, som holdes i konstant bevægelse af det langsomt gående røreaggregat. Der blandes til ensartethed.

Afslutning (C ⇒ (A + B)): Fytase (01), opført under Vandfase II, iblandes, hvorefter 02 og til sidst 03 opløses. Præparatet er nu klart til brug.

Komponent 02 og 03 kan i visse tilfælde tilsættes i vandfase I.

Komm ntarer til formul ringen

Fytasen skal indgå som en del af vitaminblandingen. Dette fremkalder den uønskede effekt, at indholdet af det ellers vandopløselige natriumriboflavin-fosfat, når det foreligger over en vis koncentration, fælder ud som riboflavin. Processen, der er irreversibel under de her fastlagte betingelser, vil gøre færdigvaren kassabel. Hastigheden, hvormed udfældningen finder sted, afhænger først og fremmest af koncentrationen af nævnte komponenter.

Urea (carbamid) anvendes her som et opløselighedsfremmende hjælpestof for riboflavin og optimeres til den respektive formulering (forskrift). Med dosering af riboflavin under et vist niveau er det ikke altid påkrævet at tilsætte urea; her kan det være tilstrækkeligt med den deklarerede mængde nicotinamid (= niacinamid). Det er i øvrigt velkendt, at dette vitamin af B-gruppen ligeledes besidder opløselighedsfremmende egenskaber. Når urea finder anvendelse i den aktuelle sammenhæng, vil det indgå med 2-10% relateret til vægt af samlet produkt.

Hvis folinsyre indgår i en formulering, behandles det på samme måde som riboflavin. Stoffet er normalt uopløseligt i vand med neutralt pH, men kan opløses i basisk miljø, hvor det imidlertid er kemisk ustabilt. Nicotinamid og urea virker begge opløselighedsfremmende.

Urea er valgt, da det foreligger i en acceptabel renhedsgrad og samtidigt findes rimeligt billigt til formålet. Desuden er der ikke påvist negativ stabilitet over for de øvrige aktive indholdsstoffer, ligesom det i de anførte koncentrationer må anses for harmløst til indvortes brug.

Flydende vitaminforblanding, påsprøjt t fter pelletering

I efterfølgende omtale af resultater fra påsprøjtning af flydende vitaminforblanding på kølede foderpiller er slagtekyllingforsøgene udeladt, da den flydende vitaminforblanding i disse forsøg blev tilført via drikkevandet. Teknisk og ernæringsmæssigt er der dog intet til hinder for at påsprøjte flydende vitaminforblanding på fjerkræfoder og alle andre pelleterede fodertyper.

Fra indledende undersøgelser haves en stærk formodning om de viste vitamintab ved foderfremstilling, og det blev derfor besluttet at undersøge, om flydende vitaminforblanding kunne påsprøjtes foderpiller efter køling med et bedre genfindingsresultat - og dermed en bedre overensstemmelse mellem tilsat vitamin og vitamin tilført dyrene. Ligeledes blev det besluttet at gennemføre produktionsforsøgene med faldende dosering af flydende vitaminforblanding sammenlignet med tør vitaminforblanding, med henblik på at fastlægge en eventuel minimumdosering, hvor dyrene reagerede via deres foderudnyttelse eller tilvækst.

I forsøget svarede den højeste dosering af flydende forblanding til doseringen af tør vitaminforblanding til kontrolfoderet (index 100 svarende til norm). Vitamindoseringen til de 4 øvrige forsøgsblandinger blev reduceret til følgende index: 85, 70, 55 og 40. Den eneste forskel på kontrol og forsøgsfoder var forblandingstypen og tilsætningsmåden.

Alle blandinger blev doseret med 2 kg pr. tons foder.

Tabel 1. Slagtesvinefoder. Genfindingsprocent r for vitamin r i forblandinger og piller ved produktion.

	Tør forblanding	Foderpiller m. tør forblanding	Flydende forblanding	Foderpiller m. flydende forblanding
Vitamin-A	79	58	83	77
Vitamin-E	92	68	103	85
Vitamin-K_3	94	4	82	32
Vitamin-B_1	60	39	70	82
Vitamin-B_2	93	74/12	101	226/72
Vitamin-B_6	67	33	91	71
Vitamin-B_{12}	42	18	82	63
Niacin	91	48	97	90
Pantotensyre	81	57	98	-
Biotin	94	40/-19	95	132/53

Af tabel 1 fremgår, at genfindingsprocenterne for vitaminer sprøjtet på overfladen af afkølede foderpiller er meget højere end for tørvitaminer, der har gennemgået foderfremstillingsprocessen. Man må imidlertid også konstatere, at genfindingsprocenterne ligger under de påsprøjtede mængder, med stor variation fra vitamin til vitamin. Undersøgelsen klarlægger ikke, om der er tale om et egentligt vitamintab på grund af kemiske reaktioner ved vitaminernes kontakt med pillernes overflade, eller om der er tale om et analyseproblem.

Vitaminerne K_3, B_{12} og biotin havde meget lave genfindingsprocenter efter tør tilsætning. Ved flydende påsprøjtning har K_3 og biotin stadig lave genfindingsprocenter, men genfindingen er dog 20 til 25 procentpoint højere. For vitamin-B_{12} er genfindingen 30 til 40 procentpoint højere. For vitamin-B_1

tyder resultater på en genfinding omkring 80 procent, hvilket er 35 til 40 procentpoint højere end ved tørvitaminer. Påsprøjtning af vitamin-B_2, -B_6 og niacin viser genfindingsprocenter, der er 30 til 40 procentpoint højere end ved anvendelse af tørvitaminer. Vitaminerne A og E har altid påkaldt sig den største interesse ved analytisk efterkontrol af foderblandingens vitaminindhold - gennemsnitsresultaterne af nærværende afprøvning viser ca. 20 procentpoint bedre genfinding for vitamin-A ved påsprøjtning og ca. 15 procentpoint for vitamin-E.

De forbedrede genfindingsprocenter for vitaminerne efter påsprøjtning har selvfølgelig betydning for dyrenes vitaminforsyning, og de er heller ikke uden betydning i relation til offentlig foderstofkontrol. Den væsentligste erkendelse er imidlertid, at vitamindoseringen ved flydende påsprøjtning kan reduceres uden at reducere dyrenes forsyning med tilsatte vitaminer.

Lagerstabilitet

Genfindingsprocenterne for vitaminer tilsat via tør vitaminforblanding eller påsprøjtet afkølede piller umiddelbart efter produktion kan ikke stå alene - lagerholdbarheden af vitaminerne er af stor betydning for såvel foderstofindustrien som husdyrproducenten.

For at følge op på dette blev der udtaget prøver af en række af de afprøvede foderpartier efter 2, 4 og 6 ugers lagring i fodersilo. Den forholdsvis korte lagertid anses for at være dækkende for hovedparten af foderblandingen anvendt i moderne husdyrproduktion.

Tabel 2: **Lagerstabilitet af vitaminer i foderpiller. Procent genfinding efter pelletering og 6 ugers lagring i forhold til indhold i melvare (naturlige vitaminer) eller deklareret tilsat (tør og flydende forblanding). Index 100 = norm. Gennemsnit af 4 produktioner.**

	Foderpiller Naturlige vitaminer		Foderpiller, tilsat tør forblanding, Index 100		Foderpiller, tilsat flydende forblanding			
					Index 100		Index 70	
	Produktion	6 uger	Produktion	6 uger	Produktion	6 uger	Produktion	6 uger
Vitamin-A, i.u./g	280/100	280/100	58	59	79	65	79	74
Vitamin-E, mg/kg	112	101	68	65	79	96	84	76
Vitamin-K_3, mg/kg	120	44	4	0/23	48	4	35	6
Vitamin-B_1, mg/kg	91	108	51	36	84	25	110	25
Vitamin-B_2, mg/kg	114	117	43	38	67	95	66/226	54/187
Vitamin-B_6, mg/kg	130	88	33	51	82	88	79	70
Vitamin-B_{12}, mg/kg	142	98	17	30	76	50	58	48
Niacin, mg/kg	102	103	48	55	84	67	90	75
Pantotensyre, mg/kg	92	88	57	68	-	-	-	-
Biotin, mg/kg	94	101	20	25	50	75	13/131	0

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Tabel 2 viser genfindingsprocenter for vitaminer ved produktion og efter 6 ugers lagring. For foderpillerne, der kun indeholder naturlige vitaminer, er genfinding i piller sat i relation til indhold i melvaren. For de øvrige blandinger er genfindingen udtrykt i forhold til garanteret tilsat. Genfinding efter 2 og 4 ugers lagring er udeladt af pladshensyn, og fordi de ikke forrykker den generelle tendens.

Generelt kan det konstateres, at alle naturligt forekommende vitaminer synes at have en stor lagerstabilitet.

Vitamin-A viser et svagt lagertab efter påsprøjtning, men tabet er væsentligt mindre end ventet. For vitaminerne E, B_2 og B_6 er der intet systematisk lagertab, ligesom pantotensyre tilsat via tør forblanding og i naturlig form ser ud til at være meget stabilt. Pantenol tilsat via flydende forblanding formodes også at være et stabilt vitamin. Det genfundne vitamin-K_3 efter flydende tilsætning reduceres hurtigt under lagring og nærmer sig 0 ved 4 ugers lagring, men ved analyse efter 6 uger fandtes de viste mængder. For vitamin-B_1 er der stor lagerstabilitet for naturligt vitamin. Det høje genfundne niveau efter påsprøjtning falder i løbet af lagerperioden ned til samme niveau som fundet ved tør vitamintilsætning. For vitamin-B_{12} ses stor forskel mellem forsøgene, både hvad angår niveau og tendens. Dog vil restmængden af vitamin efter 6 ugers lagring være væsentligt højere efter flydende påsprøjtning end efter tør tilsætning. Resultatet for niacin viser en tendens til større tab ved flydende påsprøjtning end ved tør iblanding, medens resultatet for biotin næppe lader sig tolke på grund af den store datavariation. En forsigtig tolkning kunne være, at der er 20 til 30 procent tilbage efter 6 uger uanset tilsætningsområde.

Det generelle resultat af lagringsforsøget er, at lagerstabiliteten er den samme uanset tilsætningsområde. Undtagelserne er vitamin-K_3, -B_1 og må-

ske -B_{12} efter flydende påsprøjtning, som viser noget henfald. Indholdet af disse vitaminer ved lagringsforsøgets start var dog væsentligt højere efter flydende påsprøjtning end ved tør tilsætning. Såfremt der skal være rimelige mængder af vitamin-K_3 og -B_1 i foderet efter flydende påsprøjtning, skal det opfodres inden for 2 til 4 uger efter produktion.

De gennemførte forsøg viser et overraskende stort procestab af samtlige vitaminer tilsat foderet via tør vitaminforblanding. Den manglende vitamingenfinding starter allerede efter opblanding i melvaren. En række af vitaminerne taber yderligere aktivitet under ekspanderingen, ligesom der er en tendens til fortsat tab under den efterfølgende pelletering. I foderpiller ligger den gennemsnitlige genfinding for tilsat vitamin-E og pantotensyre højest med ca. 70 procent, medens de øvrige vitaminer ligger med genfindingsprocenter fra godt 50 ned til ca. 25. Vitamin-K_3 er specielt ved stort set at forsvinde.

De gennemsnitlige genfindingsprocenter for vitaminer påsprøjtet foderpiller efter køling ligger fra 15 til 60 procentenheder højere end for vitaminer tilsat i form af tør vitaminforblanding (tabel 1).

Da samtidig lagerstabiliteten for vitaminerne ved op til 4 ugers lagring af foderblandingerne generelt er den samme uanset påføringsmåde, tilføres dyrene betydeligt højere vitaminmængder ved samme vitamindosering til foderet, når vitaminerne påsprøjtes som flydende forblanding.

Indholdet af naturlige B vitaminer viser sig at være overordentligt processtabilt, endda med en tendens til stigende indhold under foderfremstillingsprocessen. Indholdsniveauet taget i betragtning er det ikke underligt, at en foderblanding sjældent eller aldrig falder ved efterkontrol for indhold af B vitaminer. Det samme gør sig i væsentlig udstrækning gældende for vita-

min-E. Vitamin-A og specielt -K_3 har ikke denne naturlige opbakning, og da begge vitaminer i syntetisk form er noget eller meget procesfølsomme, er det indlysende, at efterkontrol her vil afsløre mange dumpere.

Genfindingsprocenter i piller for vitaminer tilsat via tør vitaminforblanding viser, at der er en meget dårlig sammenhæng mellem det deklarerede vitaminindhold (tilsat) i foderblandinger og den mængde, som når frem til dyrene. Overensstemmelsen er væsentligt bedre, når vitaminerne påsprøjtes foderpillerne. På denne baggrund var det nærliggende at undersøge, om dyrene i produktionsforsøg reagerede på reduceret vitamintilsætning via flydende forblanding påsprøjtet de kølede foderpiller.

Resultaterne af produktionsforsøgene viste, at slagtesvin ikke reagerede på en reduktion af vitamintildelingen via flydende påsprøjtning på 60 procent af den normerede tørvitamintilsætning.

De gennemførte forsøg viser, at en betydeligt højere procentdel af de tilsatte vitaminer når frem til dyrene, når vitaminforblandingen påsprøjtes færdigfoderet eller doseres gennem drikkevandet. Dyrenes vitaminbehov kan altså dækkes med en lavere tilsætning af vitaminer efter pelletering på grund af det eliminerede procestab.

Konceptet indebærer tilsætning af fytaseenzym til ekspanderet/pelleteret fjerkræfoder og svinefoder. Påsprøjtes enzymet på afkølede foderpiller sammen med vitaminforblandingen, bevares den fulde enzymaktivitet, som medfører frigørelse af 60 til 70 procent af den organisk bundne fosfor i vegetabilske foderråvarer. Herudover frigøres mindre mængder af aminosyrer, kulhydrater og mikromineraler. Som konsekvens af enzymanvendelsen og reduceret dosering af mineralsk fosfat forventes det, at fosforkon-

centrationen i gødningen samt udledningen til miljøet kan reduceres med 20 til 25 procent.

Mikromineralerne i en flydende mikromineralforblanding er 100 procent opløste, hvilket medfører, at tilgængeligheden og optageligheden for dyrene er større end ved anvendelse af de traditionelle salte. Som konsekvens kan mikromineraldoseringen til industrielt fremstillet foder reduceres med mindre udledning til miljøet til følge.

De nye produkter kan leveres i emballager, der kan håndteres med truck, og produkterne føres fra emballagen til sprayudstyret i fabrikken gennem lukkede rørsystemer. Dette medfører betydelige håndteringsmæssige og arbejdsmiljømæssige fordele for foderstofindustrien.

PATENTKRAV

1. Fremgangsmåde til fremstilling af foderpiller til dyr tilsat en forblanding, k e n d e t e g n e t ved, at forblandingen er en vitaminforblanding omfattende fedt/olie- og vandopløselige vitaminer, at foderpillerne på overfladen påsprøjtes med vitaminforblandingen, samt at foderpillerne inden påsprøjtningen har undergået en afkøling.

2. Fremgangsmåde ifølge krav 1, k e n d e t e g n e t ved, at foderpillerne efter påsprøjtning opsamles i en beholder.

3. Fremgangsmåde ifølge krav 1 og 2, k e n d e t e g n e t ved, at vitaminforblandingen er formuleret som funktion af dyrearter.

4. Fremgangsmåde ifølge ethvert af de foregående krav, k e n d e t e g n e t ved, at foderpillerne under påsprøjtning passerer en rotorspray/rotordyse.

5. Fremgangsmåde ifølge ethvert af de foregående krav, k e n d e t e g n e t ved, at foderpillerne tillige påsprøjtes en opløsning omfattende mineraler.

6. Fremgangsmåde ifølge ethvert af de foregående krav, k e n d e t e g n e t ved, at vitaminforblandingen tillige omfatter aminosyrer opløst i denne.

7. Fremgangsmåde ifølge ethvert af de foregående krav, k e n d e t e g n e t ved, at vitaminforblandingen tillige omfatter fordøjelighedsfremmende enzymer opløst i denne.

8. Fremgangsmåde ifølge ethvert af de foregående krav k e n d e t e g n e t ved, at vitaminforblandingen tillige omfatter et fytaseenzym opløst i denne.

9. Fremgangsmåde til blanding af en vitaminforblanding omfattende fedt/olieopløselige vitaminer, k e n d e t e g n e t ved, at forblandingen tillige omfatter vandopløselige vitaminer, at vandfasen omfatter propylenglycol samt EDTA og nicotinamid, hvorefter der efterfølgende tilsættes et B_2-vitamin som riboflavin og derefter natriumhydroxid (NaOH).

10. Fremgangsmåde ifølge krav 9, k e n d e t e g n e t ved, at der forud for tilsætning af B_2-vitaminet tilsættes carbamid/urea.

11. Fremgangsmåde ifølge krav 8, 9 og 10 , k e n d e t e g n e t ved, at der yderligere tilsættes saltsyre (HCl), samt at der efterfølgende tilsættes yderligere B vitaminer, fortrinsvis biotin og pyridoxinhydroklorid.

12. Fremgangsmåde ifølge krav 9, 10 og 11, k e n d e t e g n e t ved, at oliefasen omfatter A-, D- og E-vitaminer, en solubilisator samt tillige antioxidanter, hvilken blanding foregår ved et temperaturinterval på omkring 50-70°, fortrinsvis på omkring 60°.

13. Fremgangsmåde ifølge krav 9-12, k e n d e t e g n e t ved, at olie- og vandfasen sammenblandes under omrøring samt at temperaturen på vandfasen er 35-45°C.

14. Fremgangsmåde ifølge krav 9-13, k e n d e t e g n e t ved, at der tilsættes et fytaseenzym til vitaminforblandingen, hvilken forblanding fortrinsvis har en temperatur på 20-30°C.

15. Vitaminforblanding, omfattende opløselige vitaminer, k e n d e t e g n e t ved, at de oliefopløselige vitaminer omfatter A-, D- og E-vitaminer, samt at forblandingen tillige omfatter vandopløselige vitaminer, såsom K-vitamin, C-vitamin og diverse B-vitaminer.

SAMMENDRAG

Opfindelsen angår en fremgangsmåde til fremstilling af foderpiller til dyr tilsat en forblanding, som er en vitaminforblanding omfattende fedt/olie- og vandopløselige vitaminer. Foderpillerne påsprøjtes på overfladen med vitaminforblandingen, og foderpillerne har inden påsprøjtningen undergået en afkøling.

Endvidere angår opfindelsen en fremgangsmåde til blanding af en vitaminforblanding omfattende fedt/olieopløselige vitaminer, samt en vitaminforblanding omfattende opløselige vitaminer.

Herved opnås en høj genfinding af de tilsatte vitaminer.

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
16544 B PCT		
International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/DK99/00559	15.10.1999	04.11.1998
International Patent Classification (IPC) or national classification and IPC7		
A 23 K 1/00, A 23 K 1/16		
Applicant		
Leo Pharmaceutical Products LTD A/S		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 5 sheets, including this cover sheet.

   ☒ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of 2 sheets.

3. This report contains indications relating to the following items:

   - I ☒ Basis of the report
   - II ☐ Priority
   - III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - IV ☐ Lack of unity of invention
   - V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
   - VI ☐ Certain documents cited
   - VII ☐ Certain defects in the international application
   - VIII ☐ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
25.05.2000	05.03.2001
Name and mailing address of the IPEA/SE Patent- och registreringsverket Box 5055 S-102 42 STOCKHOLM Telex 17978 PATOREG-S Facsimile No. 08-667 72 88	Authorized officer Eva Johansson/EÖ Telephone No. 08-782 25 00

Form PCT/IPEA/409 (cover sheet) (January 1998)

EL2431041670S

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/DK99/00559

I. Basis of the report

1. With regard to the elements of the international application:*

☐ the international application as originally filed

☒ the description:
pages 1-17 , as originally filed
pages _____ , filed with the demand
pages _____ , filed with the letter of _____

☒ the claims:
pages _____ , as originally filed
pages _____ , as amended (together with any statement) under article 19
pages _____ , filed with the demand
pages 18-19 , filed with the letter of 05.01.2001

☐ the drawings:
pages _____ , as originally filed
pages _____ , filed with the demand
pages _____ , filed with the letter of _____

☐ the sequence listing part of the description:
pages _____ , as originally filed
pages _____ , filed with the demand
pages _____ , filed with the letter of _____

2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.
These elements were available or furnished to this Authority in the following language english which is:

☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).

☒ the language of publication of the international application (under Rule 48.3(b)).

☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

☐ contained in the international application in written form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. ☐ The amendments have resulted in the cancellation of:

☐ the description, pages _____

☐ the claims, Nos. _____

☐ the drawings, sheet/fig _____

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2 (c)).**

* *Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).*

** *Any replacement sheet containing such amendments must be referred to under item I and annexed to this report.*

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/DK99/00559

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims	1-12	YES
	Claims		NO
Inventive step (IS)	Claims		YES
	Claims	1-12	NO
Industrial applicability (IA)	Claims	1-12	YES
	Claims		NO

2. Citations and explanations (Rule 70.7)

The claimed invention relates to a method for the production of animal feed pellets with the addition of a premix, which is a vitamin premix comprising fat/oil and water-soluble vitamins. The surface of the pellets is sprayed with the vitamin premix and the feed pellets are cooled before being sprayed.

New amended claims 1-12 have been filed on 5 January 2001. The auxiliary request claims have been examined.
Claim 1 has been supplemented with the specification of the cooling temperature to be equal or less than 50^0 C, that the vitamin premix also comprises a phytase enzyme and that the pellets are collected in a container.

The technical features "phytase enzyme" and "collected in a container" do not solve the problem of the invention, which is a method for the production of a feed pellets sprayed with a vitamin premix and cooled before spraying.
It is obvious to a person skilled in the art to chose those products e.g. oil/fat or water soluble vitamins, minerals, amino acids and enzymes that are suitable for each opportunity and mix the products to a liquid premix.
It is also obvious to a person skilled in the art to collect the finished pellets in a container.

.../....

Supplemental Box
(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: V

The following documents are cited in the search report:
D1) EP 689834
D2) EP 231817
D3) EP 682874
D4) ES 2033571
D5) WO 9847389
D6) GB 1457643
D7) RU 2075946 abstract

D2) relates to a method of pelletising animal or human foods with vitamin(s) and similar substances preferably sprinkled onto finished cooled pellets. The pellets are made of foodstuffs or animal feed, mixed and heated in a pelletising process. The pellets are cooled (see col. 7 line 3-15) before the health-promoting additives such as vitamins (see example 3 line 43 and 51-56) are sprayed or rolled on the surface of the pellets (see fig 1, and col. 8).

The claimed invention only differs from the known in the definition of the temperature.
It is obvious to a person skilled in the art from reading the cited document to choose suitable vitamins and other health-promoting additives and to mix these components to a premix and spray the mixture on the pellets at an appropriate temperature. Thus, the claimed invention lacks inventive step.

D4) relates to an animal feed supplement production. Granules are made in a spray tower and then cooled to 35^0 C. The cooled granules are sprayed with a vitamin complex (consisting of different vitamins and other health-promoting additives (see col. 4) which remains fixed to the surface of the granules.

From this document is it known to cool the granules to a temperature of 35^0 C before spraying the mixture on the granules. Thus, it is obvious to a person from reading the cited documents to cool granules or pellets before spraying with a mixture.

D1) relates to a food and feed supplement comprising vitamins. A core mixture of vitamins is prepared. A basic mixture is prepared and agglomerated. After drying granules are made and sprayed with the core mixture. The core mixture contains vitamins and other health-promoting additives (see example 5 page 11-12).

..../...

Supplemental Box
(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: V

D3) relates to bioactive feed pellets comprising besides commonly used nutritionally valuable components, a bioactive ingredient such as therapeutically or prophylactically active compound, a vaccine, a pigment, a vitamin, and/or an enzyme. The bioactive ingredient is applied to the pellets in the form of a primary coated dispersion.

D5), D6) and D7) relate to among other things a vitamin premix comprising commonly used vitamins and other health-promoting additives, see particular page 7 line 15 to page 8 line 14 in D5) and example 1-5 in D6).

From the last cited documents it is known to prepare mixtures containing e.g. oil/fat and water- soluble vitamins, minerals, amino acids and enzymes.
According to what is known from the cited documents is it obvious to a person skilled in the art to mix suitable vitamins with an enzyme.
There is no information in the application that an addition of a phytase enzyme in the vitamin premix would have any special effect on the spraying process.

The claimed invention is considered to be novel and has industrial applicability but is considered to lack inventive step.

AUXILIARY REQUEST

1. Method for the production of animal feed pellets with the addition of a premix, characterized in that the premix is a vitamin premix comprising fat/oil- and water-soluble vitamins, that the surface of the feed pellets is sprayed with the vitamin premix, and in that the feed pellets are subjected to cooling before being sprayed said temperature being equal or less than 50°C and that the vitamin premix also comprises a phytase enzyme dissolved in said vitamin premix and that after being sprayed the feed pellets are collected in a container.

2. Method according to claim 1, characterized in that the vitamin premix is formulated as a function of animal species.

3. Method according to any of the foregoing claims, characterized in that the feed pellets pass a rotor-spray/rotor nozzle when being sprayed.

4. Method according to any of the foregoing claims, characterized in that the feed pellets are also sprayed with a solution comprising minerals.

5. Method according to any of the foregoing claims, characterized in that the vitamin premix also comprises amino acids dissolved in said vitamin premix.

6. Method according to any of the foregoing claims, characterized in that the vitamin premix also comprises digestibility-promoting enzymes dissolved in said vitamin premix.

7. Method for the mixing a vitamin premix comprising fat/oil-soluble vitamins, characterized in that the premix also comprises water-soluble vitamins, that the water phase comprises propylene glycol and EDTA and

nicotinamide, after which a B_2 vitamin is subsequently added such as riboflavin and thereafter sodium hydrochloride (NaOH).

8. Method according to claim 7, c h a r a c t e r i z e d in that carbamide/urea is added before the addition of the B_2 vitamin.

9. Method according to claim 7 and 8, c h a r a c t e r i z e d in that hydrochloric acid (HCl) is also added, and that further B vitamins are subsequently added, mainly biotin and pyridoxine hydrochloride.

10. Method according to claim 7, 8 and 9, c h a r a c t e r i z e d in that the oil phase comprises A, D and E vitamins, a solubilisator and also antioxidants, the mixing of which is carried out at a temperature interval of around 50-70°, preferably at around 60°.

11. Method according to claims 7-10, c h a r a c t e r i z e d in that the oil phase and the water phase are mixed together while being stirred, and that the temperature of the water phase is 35-45°C.

12. Method according to claims 7-11, c h a r a c t e r i z e d in that a phytase enzyme is added to the vitamin premix, said premix preferably having a temperature of 20-30°C.

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METHOD FOR THE PRODUCTION OF FEED PELLETS, MIX OF A PREMIX AND VITAMIN PREMIX.

The invention concerns a method for the production of animal feed pellets with the addition of a premix.

The invention also concerns a method for the mixing of a vitamin premix comprising fat/oil-soluble vitamins.

Finally, the invention comprises a vitamin premix comprising soluble vitamins.

It is known from EP-A-682874 to add vitamins to animal feeds, said vitamins existing in a fat or oil solution, by which product and by which method there should arise a smaller risk of the decomposition of the vitamin admixture. However, the publication does not disclose a method for the addition of the total physiological vitamin requirement which is necessary in connection with animal feeds, and the publication does not either state any method whereby this admixture of vitamins takes place under conditions where damage and decomposition of the individual vitamins, whether both fat and water soluble, is minimised to the greatest possible degree.

It is the object of the present invention to provide a method whereby the above-mentioned problems are overcome, and whereby the percentage of recovery for the added vitamins is very high, whereby overdosing is avoided, and whereby it is possible to effect a spraying of liquid premixes on the feed pellets, so that investments in larger plants for the handling of premixes following the dry admixture method is made superfluous.

It is also the object by this method to ensure that the processing and storage stability is optimised, so that the least possible decomposition of

the vitamins occurs after these have been sprayed on to the industrially-produced feeds.

This object is achieved with a method of the kind disclosed in the preamble, and also where the premix is a vitamin premix comprising fat/oil and water-soluble vitamins, where the surface of the feed pellets is sprayed with the vitamin premix, and where the feed pellets have been subjected to cooling before the spraying.

By carrying out a spraying of the feed pellets when these are produced, and by also carrying out a suitable cooling to a maximum of 50°C and an optimum temperature level of 25-35°C, it is possible to effect a subsequent spraying of the surface of the pellets with the vitamin premix, which comprises both fat/oil- and water-soluble vitamins, whereby it is ensured that the feed pellets receive an optimum content of vitamins for the relevant species of animals, whether these be pigs or chickens etc. By this method, a far higher recovery of the added vitamins is achieved than with a corresponding dosing via a dried vitamin premix.

The substantially improved recovery percentage for the vitamins after spraying is of importance for the animals' vitamin supply, and is thus also significant in relation to the feed control authorities. Consequently, it is important that the dosing of vitamins by liquid spraying can be reduced without reducing the animals' supply of added vitamins.

By providing a method according to the invention and as further disclosed in claim 2, it is achieved that the feed pellets are stored at a relatively low concentration of oxygen, whereby the decomposition is minimised and the storage stability is optimised. A storage time of 2-6 weeks, which is a relatively short but nevertheless a realistic storage time, thus results in the vitamin stability and the recovery being surprisingly great.

By providing a method according to the invention and as further disclosed in claim 3, the possibility is established for a regulation of the finish-produced feed pellets with the vitamin premix with which they are sprayed, all depending on whichever animal may be involved. In other words, this means that where the feed is to be used, for example, for chickens, the pellets can be sprayed with vitamins comprising the C vitamin.

By providing a method according to the invention, and as further disclosed in claim 4, a good spraying on the individual pellets is achieved, so that any section of the pellets will give rise to substantially the same amount of concentration of the premix with which they are sprayed.

By providing a method according to the invention and as further disclosed in claim 5, it is achieved that the feed pellets also come to comprise the minerals which may be relevant for the individual animals.

By providing a method according to the invention and as further disclosed in claim 6, the possibility is achieved of adding further nutrients such as lysine, mhehionine, threonine, leucine and isoleucine.

By providing a method according to the invention and as further disclosed in claim 7, the possibility is achieved for the addition of enzymes such as carbohydrate- and protein-spliced enzymes.

By providing a method according to the invention and as further disclosed in claim 8, a saving is achieved in the amount of added phosphor, and which otherwise means that the selected B vitamin should predominantly be riboflavin and not sodium riboflavin phosphate.

As disclosed in the preamble, the invention also concerns a method for the mixing of the actual vitamin premix which is used during the spraying, and

where the premix also includes water-soluble vitamins, that the water phase comprises propylene glycol and EDTA and nicotinamide, after which there is subsequently added a B_2 vitamin such as riboflavin and sodium hydroxide (NaOH).

With such a premix comprising the above-mentioned components, the possibility is provided of dissolving the riboflavin, which is important in that riboflavin is not only advantageous from the point of view of cost, but also necessary to use as B_2 vitamin if phytase enzyme is to be added later.

By providing a method according to the invention and as further disclosed in claim 10, the possibility is provided of being able to add greater amounts of B_2 vitamin such as riboflavin.

By providing a method according to the invention and as further disclosed in claim 11, a reduction in the pH is achieved, so that the added riboflavin or sodium riboflavin phosphate is not ruined by the high pH which is established by the addition of sodium hydroxide (NaOH).

By providing a method according to the invention and as further disclosed in claim 12, an expedient composition of the oil phase is achieved.

By providing a method according to the invention and as further disclosed in claim 13, a homogenous substance of the premix is achieved during stirring.

By providing a method according to the invention and as further disclosed in claim 14, it is achieved that the amount of phosphate secreted by the animals is reduced. Moreover, the solubility of the riboflavin is ensured due to the presence of urea.

The temperature of 20-30° in the premix can be achieved by the addition of cold water before the addition of the phytase.

The invention also concerns a vitamin premix such as that disclosed in claim 15.

The invention will be explained in more detail with reference to the following example:

LIQUID VITAMINS

Code	*Raw material*	*Weight in grams*	
01-A	Vitamin-A-acetate 2.5 mill. IU/g	0.930	Oil phase
02-A	Vitamin-D_3-oil 4.0 mill. IU/g	0.060	-
03-A	Vitamin-E-oil 97%	39.950	-
04-A	Ethoxyquin	2.000	-
05-A	Bredol 694	50.000	-
01-B	Choline chloride 75% solution*	0	Water phase I
02-B	Vitamin-B_{12} 2% solution	0.610	-
03-B	Panthenol	5.100	-
04-B	Biotin 2% to liquid vitamins	1.040	-
05-B	Pyridoxine hydrochloride	1.240	-
06-B	Sodium riboflavin phosphate	0.590	-
07-B	Sodium hydroxide solution 34° Bé	2.600	
08-B	Riboflavin 97%	0.590	-
09-B	Hydrochloric acid 10%	2.800	-
09-B	Folinic acid 100%*	0	-

Code	*Raw material*	*Weight in grams*	
10-B	Urea 46 (Carbamide)	50.000	-
11-B	Nicotinamide (=niacinamide)	9.050	-
12-B	Potassium sorbate (preservative agent)	2.000	-
13-B	Tetracemindinsodium (complex binder)	2.000	-
14-B	Propylene glycol (dissolution-promoting and preservative agent)	100.000	-
15-B	Water	666.550	-
01-C	Phytase solution 5000 FTU/g	60.000	Water phase II
02-C	Thiamine hydrochloride	0.990	-
03-C	Menadione sodium bisulphate 100%	1.900	-
	Total – grams:	1000.000	

* relevant for poultry.

Before the 07 base is added, the pH is approx. 6, after which the base addition results in a pH increase to approx. 10-11, which is necessary in order for the riboflavin (08) to be dissolved. Component 09 is then added to effect a reduction of the pH to around 8, in that the substance 08 is ruined by a pH which is too high. By carrying out a mixing as described, it is achieved that all vitamins are present in the desired concentrations, and without destruction of these taking place.

Generally, the carbamide weight percentage interval of the total amount will lie in the interval 2-10%, mainly around 5%.

If the sodium riboflavin phosphate is omitted, the necessary amount of carbamide in the example will increase to 50-60 grams in order to ensure the solubility of the riboflavin.

The solution itself consisting of both water-soluble as well as oil-soluble vitamins is subsequently sprayed on the feed pellets for animals. This spraying can be effected before the pellets are stored in silos, or spraying can be effected when the pellets are loaded into transport containers. Both solutions enable a differentiation to be made between the different animals, e.g. pigs vs. chickens, and differentiation in accordance with customer requirements.

It has shown surprisingly that when this spraying of the vitamins on the pellets is carried out, no destruction or precipitation of the vitamins occurs, which means that the pellets can be considered to be particularly stable. The reason why no destruction occurs can possibly be that the pellets are stored in silos and containers in which there is a low percentage of oxygen.

Production principle

The final preparation consists of an oil phase comprising the so-called fat-soluble vitamins mixed up with an adjusted amount of a suitable solubility product – here, use is made of Bredol 694. This mixture, preheated to 55-65°C, is poured/pumped in an adjusted flow down into the water phase containing the water-soluble components and with a temperature set at 40-45°C. The distribution of the oil phase with subsequent dissolution in the water phase, the actual solubilising process, must take place under constant stirring and at a suitably calm pace.

The mixing procedure

The oil phase (code A): The auxiliary material 05 is added one at a time and in the given sequence, the vitamins 01, 02 and 03 and the antioxidant 04. The components are preheated in order to achieve a suitable consistency for handling. Mixing is effected to uniformity. Hereafter, the mixture will continue to be homogeneous.

The water phase I (code B): Measured amounts of water 15 (preheated to approx. 45°C) are mixed with 14, in which 13, 12, 11 and 10 are dissolved in this order. Thereafter, 08 is added to the mixture (insoluble in water at this process stage). When the material appears to be distributed uniformly in the water phase, the base 07 is added. Upon conclusion of the dissolution of 08, the acid 09 is added and thereafter 06, 05, 04,03 and 02 one at a time and in this order. The product appears as a slightly opalescent liquid.

Solubilising phase (A ⇒ B): The oil phase A is now pumped in a thin and continuous stream down into the water phase I, which is held in constant movement by the slowly-moving stirring unit. Mixing is effected to uniformity.

Completion (C ⇒ (A + B)) Phytase (01), performed under water phase II, is mixed in, after which 02 and finally 03 are dissolved. The preparation is now ready for use.

In certain cases, the components 02 and 03 can be added in water phase I.

Comments on the formulation

The phytase must be included as a part of the vitamin mixture. This gives rise to the undesired effect that the content of the otherwise water-soluble sodium riboflavin phosphate, when it exists above a certain concentration, is precipitated as riboflavin. The process, which is irreversible under the conditions determined here, will render the finished product useless. The speed at which the precipitation takes place depends first and foremost on the concentration of said components.

Urea (carbamide) is used here as a solubility-promoting material for riboflavin and is optimised for the respective formulation (model). With dosing of riboflavin below a certain level, it is not always necessary to add urea, in that it can be sufficient here with the declared amount of nicotinamide (= niacinamide). It is otherwise well-known that this vitamin of the B-group similarly possesses solubility-promoting characteristics. When the use of urea is found in the actual context, it will form part with 2-10% related to weight of the total product.

If folinic acid is included in a formulation, this is handled in the same way as riboflavin. The material is normally insoluble in water with neutral pH, but can be dissolved in basic solutions where, however, it is chemically unstable. Both nicotinamide and urea serve to promote solubility.

Urea has been selected for the reason that it exits in an acceptable degree of purity, and at the same time it is found to be reasonably cheap for the purpose. Moreover, no negative stability has been shown with regard to the remaining active material contents, and in the stated concentrations it must be considered to be harmless for internal use.

Liquid vitamin mixture, sprayed on after pelleting.

In the following discussion of the results from the spraying of liquid vitamin premix on cooled feed pellets, the tests with broilers have been omitted, the reason being that the liquid vitamin premix in these tests was introduced via drinking water. However, technically and from the point of view of nutrition, there is nothing to prevent the spraying of liquid vitamin premixes on poultry feeds and all other pelleted types of feeds.

Preliminary investigations gave rise to a great deal of conjecture concerning the shown vitamin loss in the production of animal feeds, and therefore it was decided to examine whether feed pellets could be sprayed with liquid vitamin premixes after cooling with a better recovery result – and herewith a better correlation between the added vitamin and the vitamin supplied to the animals. Similarly, it was decided to carry out production tests with decreasing dosage of liquid vitamin premix compared with dry vitamin premix, the object being to determine a possible minimum dosage where the animals reacted via their feedstuff utilisation or growth.

In the trial, the highest dosing of liquid premix corresponded to the dosing of dry vitamin premix to the control feed (index 100 corresponding to standard). The dosing of vitamin to the 4 remaining trials mixtures was reduced to following index: 85, 70, 55 and 40. The only difference in the control and trial feed was the premix type and the method of addition.

All mixtures were dosed with 2 kg. per ton feedstuff.

Table 1. Feeds for porkers. Recovery percentages for vitamins in premixes and pellets at production.

	Dry premix	Feed pellets with dry premix	Liquid premix	Feed pellets with liquid premix
Vitamin-A	79	58	83	77
Vitamin-E	92	68	103	85
Vitamin-K_3	94	4	82	32
Vitamin-B_1	60	39	70	82
Vitamin-B_2	93	74/12	101	226/72
Vitamin-B_6	67	33	91	71
Vitamin-B_{12}	42	18	82	63
Niacin	91	48	97	90
Pantothenic acid	81	57	98	-
Biotin	94	40/-19	95	132/53

From Table 1 it will be seen that the recovery percentages for vitamins sprayed on the surface of cooled feed pellets are very much higher than for dry vitamins which have gone through the feed production process. However, it must also be ascertained that the recovery percentages lie below the sprayed-on amounts, with great variation from vitamin to vitamin. The investigation does not clarify the question of whether there is an actual vitamin loss due to chemical reactions upon the contact of the vitamins with the surface of the pellets, or whether an analysis problem is involved.

The vitamins K_3, B_{12} and biotin had very low recovery percentages after dry addition. With liquid spraying, K_3 and biotin still have low recovery percentages, but nevertheless the recovery is 20 to 25 percentage points higher. For vitamin B_{12}, the recovery is 30 to 40 percentage points higher.

For vitamin B_1, the results indicate a recovery of around 80 percent, which is 35 to 40 percentage points higher than with dry vitamins. The spraying-on of vitamin B_2, B_6 and niacin shows recovery percentages which are 30 to 40 percentage points higher than with the use of dry vitamins. The vitamins A and E have always attracted the greatest interest in analytical post-control of the vitamin contents of the feed mixture – the average results of the present tests show approximately 20 percentage points better recovery for sprayed vitamin A and approx. 15 percentage points for vitamin E.

The improved recovery percentages for the vitamins after spraying are naturally of importance for the animals' vitamin supply, and they are not without significance in relation to the feed control authorities. However, the most important acknowledgement is that the vitamin dosing by liquid spraying can be reduced without reducing the animals' supply of the added vitamins.

The storage stability

The recovery percentages for vitamins added via dry vitamin premixes or sprayed on cooled pellets immediately after production can not stand alone – the storage durability of the vitamins is of great importance both for the feed industry and the animal breeder.

To follow up on this, samples were taken of a number of the tested feed portions after storage for 2, 4 and 6 weeks in a feed silo. The relatively short storage time is considered to be appropriate for most of the feed mixtures used in modern animal breeding.

Table 2: Storage stability of vitamins in feed pellets. Percentage recovery after pelleting and 6 weeks storage in relation to meal product (natural vitamins) or declared additives (dry and liquid premix).

Index 100 = standard. Average of 4 productions.

	Feed pellets Natural vitamins		Feed pellets with dry pre-mix added, Index 100		Feed pellets with liquid premix added			
					Index 100		***Index 70***	
	Production	6 weeks	Production	6 weeks	Production	6 weeks	Production	6 weeks
Vitamin-A, i.u./g	280/100	280/100	58	59	79	65	79	74
Vitamin-E, mg/kg	112	101	68	65	79	96	84	76
Vitamin-K_3, mg/kg	120	44	4	0/23	48	4	35	6
Vitamin-B_1, mg/kg	91	108	51	36	84	25	110	25
Vitamin-B_2, mg/kg	114	117	43	38	67	95	66/226	54/187
Vitamin-B_6, mg/kg	130	88	33	51	82	88	79	70
Vitamin-B_{12}, mg/kg	142	98	17	30	76	50	58	48
Niacin, mg/kg	102	103	48	55	84	67	90	75
Pantothenic acid mg/kg	92	88	57	68	-	-	-	-
Biotin, mg/kg	94	101	20	25	50	75	13/131	0

Table 2 shows recovery percentages for vitamins at production and after storage for 6 weeks. For feed pellets which only contain natural vitamins, the recovery in pellets is set in relation to the contents in the meal product. For the remaining mixtures, the recovery is expressed in relation to the guaranteed addition. Recovery after 2 and 4 weeks' storage is omitted out of regard for space, and because they do not influence the general tendency.

In general it can be ascertained that all naturally occurring vitamins would appear to have a greater storage stability.

Vitamin A shows a slight storage loss after spraying, but the loss is considerably less than expected. For the vitamins E, B_2 and B_6, there is no systematic storage loss, and pantothenic acid added via dry premix and in natural form appears to be very stable. Pantenol applied via liquid premix can also be assumed to be a stable vitamin. The vitamin K_3 retained after liquid application is quickly reduced during storage and it approaches 0 at 4 weeks' storage, but the amounts shown were found upon analysis after 6 weeks. For vitamin B_1 there is great storage stability for natural vitamin. During the storage period, the high recovery level after spraying falls down to the same level as found with dry vitamin addition. For vitamin B_{12}, great difference is seen between the tests, both with regard to level and tendency. However, the amount of vitamin remaining after storage for 6 weeks will be considerably higher after liquid spraying than after dry addition. The result for niacin shows a tendency towards greater loss with liquid spraying than with dry addition, while the result for biotin hardly permits itself to be interpreted due to the great variation in data. A guarded interpretation could be that there is 20 to 30 percent remaining after 6 weeks regardless of the method of application.

The general result of the storage test is that the storage stability remains the same regardless of the method of application. The exceptions are vitamin K_3, B_1 and perhaps B_{12}, which after liquid spraying shows some decrease. However, the contents of these vitamins at the start of the storage test were considerably higher after liquid spraying than with dry addition. If there are to be reasonable amounts of vitamin K_3 and B_1 in the feed after liquid spraying, the feed must be used within 2 to 4 weeks after production.

The tests carried out show a surprisingly great process loss of all vitamins added to the feed via dry vitamin premix. The lacking vitamin recovery starts already after admixture with the meal product. A number of vitamins lose further activity during expansion, and there is a tendency towards continued loss during the subsequent pelleting. In feed pellets, the average recovery for added vitamin E and pantothenic acid is highest with approx. 70 percent, while the recovery percentages for the remaining vitamins lie a good 50 to approx. 25 percent down. Vitamin K_3 is particular in that it more or less disappears.

The average recovery percentages for vitamins sprayed on feed pellets after cooling lies from 15 to 60 percentage points higher than for vitamins added in the form of dry vitamin premix (table 1).

Since at the same time the storage stability for the vitamins with up to 4 weeks' storage of the feed mixtures is generally the same regardless of the method of application, the animals are provided with considerably higher amounts of vitamin at the same vitamin dosing to the feed when the vitamins are sprayed on as liquid premix.

The content of natural B vitamins proves to be extremely stable during processing, and even with a tendency towards increasing content during the feed production process. With the content level taken into con-

sideration, it is not curious that a feed mixture seldom or never falls at the post-control for the content of B vitamins. The same applies to a considerable extent regarding vitamin E. Vitamin A and especially vitamin K_3 do not have this natural back-up, and since both vitamins in synthetic form are somewhat or very sensitive to processing, it is obvious that post-control here will reveal many failures.

The recovery percentages in pellets for vitamins added via dry vitamin premixes show that there is a very poor correlation between the declared vitamin content (added) in the feed mixtures and that amount which reaches the animals. The relationship is considerably better when the pellets are sprayed with vitamins. In light of this, it was natural to carry out an investigation into whether the animals in production tests reacted to reduced vitamin addition via liquid premix sprayed on the cooled feed pellets.

The results of the production tests showed that porkers did not react to a reduction of vitamin allocation via liquid spraying of 60 percent of the prescribed dry vitamin addition.

The tests performed show that a considerably higher percentage of the added vitamins reach the animals when the vitamin premix is sprayed on the finished feed or dosed via the drinking water. Consequently, due to the elimination of the process loss, the animals' vitamin requirements can be covered with a smaller addition of vitamins after pelleting.

The concept involves the addition of phytase enzyme to expanded/pelleted poultry feeds and pig feeds. When the enzyme is sprayed on cooled feed pellets together with the vitamin premix, the full enzyme activity is retained, which gives rise to the release of 60 to 70 percent of the organically-bound phosphor in vegetable feed raw materials. Furthermore, smaller amounts of amino acids, carbohydrates and micro-minerals are released. As a con-

sequence of the use of enzymes and reduced dosing of mineral phosphate, it is expected that the concentration of phosphor in the manure and the discharge to the environment can be reduced by 20 to 25 percent.

The micro-minerals in a liquid micro-mineral mixture are 100 percent dissolved, the result being that the accessibility to and the absorption by the animals is greater than with the use of traditional salts. As a consequence, the dosing of micro-minerals to industrially-produced feeds can be reduced, with subsequently less discharge to the environment.

The new products can be delivered in packaging which can be handled by truck, and the products are conveyed from the packaging to the spraying equipment in the feed mill through closed piping systems. This results in considerable advantages for the animal feed industry, both with regard to handling as well as the working environment.

CLAIMS

1. Method for the production of animal feed pellets with the addition of a premix, characterized in that the premix is a vitamin premix comprising fat/oil- and water-soluble vitamins, that the surface of the feed pellets is sprayed with the vitamin premix, and in that the feed pellets are subjected to cooling before being sprayed.

2. Method according to claim 1, characterized in that after being sprayed the feed pellets are collected in a container.

3. Method according to claim 1 and 2, characterized in that the vitamin premix is formulated as a function of animal species.

4. Method according to any of the foregoing claims, characterized in that the feed pellets pass a rotor-spray/rotor nozzle when being sprayed.

5. Method according to any of the foregoing claims, characterized in that the feed pellets are also sprayed with a solution comprising minerals.

6. Method according to any of the foregoing claims, characterized in that the vitamin premix also comprises amino acids dissolved in said vitamin premix.

7. Method according to any of the foregoing claims, characterized in that the vitamin premix also comprises digestibility-promoting enzymes dissolved in said vitamin premix.

8. Method according to any of the foregoing claims, characterized in that the vitamin premix also comprises a phytase enzyme dissolved in said vitamin premix.

9. Method for the mixing a vitamin premix comprising fat/oil-soluble vitamins, c h a r a c t e r i z e d in that the premix also comprises water-soluble vitamins, that the water phase comprises propylene glycol and EDTA and nicotinamide, after which a B_2 vitamin is subsequently added such as riboflavin and thereafter sodium hydrochloride (NaOH).

10. Method according to claim 9, c h a r a c t e r i z e d in that carbamide/urea is added before the addition of the B_2 vitamin.

11. Method according to claim 8, 9 and 10, c h a r a c t e r i z e d in that hydrochloric acid (HCl) is also added, and that further B vitamins are subsequently added, mainly biotin and pyridoxine hydrochloride.

12. Method according to claim 9, 10 and 11, c h a r a c t e r i z e d in that the oil phase comprises A, D and E vitamins, a solubilisator and also antioxidants, the mixing of which is carried out at a temperature interval of around 50-70°, preferably at around 60°.

13. Method according to claims 9-12, c h a r a c t e r i z e d in that the oil phase and the water phase are mixed together while being stirred, and that the temperature of the water phase is 35-45°C.

14. Method according to claims 9-13, c h a r a c t e r i z e d in that a phytase enzyme is added to the vitamin premix, said premix preferably having a temperature of 20-30°C.

15. Vitamin premix comprising soluble vitamins, c h a r a c t e r i z e d in that the oil-soluble vitamins comprise A, D and E vitamins, and that the premix also comprises water-soluble vitamins such as K vitamin, C vitamin and various B vitamins.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/DK 99/00559

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: A23K 1/00, A23K 1/16
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: A23K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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X	EP 0231817 A2 (GEBRüDER BüHLER AG), 12 August 1987 (12.08.87) --	1-14
Y	EP 0682874 A2 (EWOS AKTIEBOLAG), 22 November 1995 (22.11.95) --	1-14
Y	ES 2033571 A1 (GERMAOL, S.L.), 16 March 1993 (16.03.93) --	1-14

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" erlier document but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
21 February 2000	28 -02- 2000
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INTERNATIONAL SEARCH REPORT

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PCT/DK 99/00559

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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INTERNATIONAL SEARCH REPORT

Information on patent family members

02/12/99

International application No.
PCT/DK 99/00559

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